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10 April 1957

CONFIDENTIAL

MEMORANDUM FOR: Office of Logistics/Procurement Division/Contract Branch

SUBJECT: Request for Additional Work under Task C of Contract
ND-86 with [REDACTED]

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REFERENCE: Memorandum for OL/PD/CB from C/TDS/ED dated
25 April 1956

1. The referenced memorandum requested initiation of Task C under Contract ND-86 with [REDACTED] for a research program directed toward the development of a prototype hydrogen generator. Twenty-one thousand ninety dollars was allocated for Phase I of the program which consisted of relatively small-scale studies of the requirements for an experimental hydrogen generator and the preliminary design of an experimental full-scale hydrogen generator. The first phase of this program has been satisfactorily completed. It is now desirable that action be initiated for Phase II of the program for research directed toward the development and evaluation of a prototype full-scale hydrogen generator in accordance with the contractor's proposal attached hereto.

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2. It is requested that the contractor be given approval to produce two experimental generator units at a cost of approximately \$6,500.00 by a letter accompanying the contract. By memorandum dated 27 March 1957 we forwarded to OL/PD/CB requisition DMS-313-27-3137-57 for the purchase of the sodium borohydride which is to be shipped to Nashville for use in the performance of this work. The balloons for use in the full-scale experiments will be provided through this office at the appropriate time.

3. It is therefore requested that Task C under Contract ND-86 be amended in the amount of \$18,285.00 and that the completion date of this task be extended from 29 January 1957 until eight months from the date of receipt of the supplement to proceed with Phase II of this program. Charges for this additional amount are to be made against Allotment Number 7-2528-10.

4. Contract ND-86 is an Agency sterile contract. Task C is also Agency sterile; however Government interest may be shown. The item, except in its full scale testing phase, will be unclassified. Agency interest in all work and material under the contract is classified Secret and may be divulged only on a need-to-know basis to appropriate security approved personnel.

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5. Further information concerning this program may be obtained
from the project engineer, [redacted] Room 210, West Outbuilding, 25X1
[redacted] 25X1

[redacted] *TCW*
Chief
TES/Engineering Division 25X1

Attachments:

TES-913-07-3119-57

Cont'r Proposal dtd 3/27/57

APPROVED FOR THE COLLECTION OF FUNDS:

Research Director

Date

ED/P/TSS/LB/ME

Distribution:

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HYDROGEN GENERATOR

In replying please address:

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March 27, 1957

Dear Sir:

Enclosed is our proposal for an additional eight-month period of research on the development of a hydrogen generator. It is based on recent discussions with your technical representatives.

In connection with the performance of the proposed research, it is our understanding that your technical representatives will furnish the sodium borohydride needed and also one or two balloons for use in the full-scale experiments.

To facilitate the prosecution of the proposed program, it is recommended that the contract contain express approval for the procurement of two experimental generator units at a cost of approximately \$6,500. As an alternative, the Contracting Officer's approval might be granted in an accompanying letter.

We are looking forward to working together on this proposed program. If you should have any questions regarding our proposal, please let us know. Any inquiries of a contractual nature may be directed to

[redacted] at Extension 875.

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Very truly yours,

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[redacted]
Director

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[redacted]
Enclosure
In Duplicate

EXTENSION PROPOSAL
on
THE DEVELOPMENT OF A HYDROGEN GENERATOR

Introduction and Objective

This proposal presents Phase II of a research program directed toward the development of a prototype hydrogen generator. Phase I of this program was conducted under Task Order No. C of our current contract and was devoted to relatively small-scale studies of the requirements for an experimental hydrogen generator capable of generating enough hydrogen to provide 250 lb of lift at sea level (about 3,500 cu ft of hydrogen) over a period of 45 to 60 minutes. Phase I included a literature search, an analysis of the most desirable systems compatible with the Sponsor's specifications for the generator of interest, a laboratory investigation of the characteristics of the hydrogen-generating reaction, and the preliminary design of an experimental full-scale hydrogen generator.

The objective of Phase II of the research program, as proposed here, would be to conduct research directed toward the development and evaluation of a prototype full-scale (about 3,500 cu ft) hydrogen generator, with the Sponsor's stated specifications being goals for the proposed experimental device. In view of the high cost of the basic chemical (sodium borohydride), the evaluation portion of the proposed program would include a series of 1/5-scale studies, and then two or three full-scale studies.

Conclusions of Phase I (Task Order No. C) Study

The research performed under Phase I showed that the cobalt

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chloride - sodium borohydride hydrolysis system would probably most satisfactorily meet the requirements of the Sponsor. On the basis of this previous research, the below-indicated details relative to the hydrolysis reaction in and the proposed design for the prototype generator were tentatively arrived at:

Pool dimensions	1-1/2 ft in depth x 50 sq ft of surface area
Total solution	Approximately 560 gal
Sodium hydroxide	0.2 lb
Sodium borohydride (98 %)	100 lb
Capacity	3,700 cu ft H ₂ in 45 to 60 min
Cobalt chloride	2.4 lb in approximately 10 gal H ₂ O
Initial temperature	72 F (22 C)
Highest temperature of off-gas	130 F (54 C)

The Phase I study showed that, under adiabatic conditions, this reaction system may be scaled to substantially any size. The largest size of experimental generator used in the Phase I study produced about 36 cu ft of hydrogen; this was a vertical cylinder 10 in. in diameter and 1-1/2 ft in height. (Scaling was reduced to a "one-dimensional problem" by keeping the depth of solution constant at 1-1/2 ft.) Because the reaction in this experimental generator, as well as in the smaller experimental units, was approximately adiabatic, it was concluded that there would probably be no major problems in scaling up experimental generators beyond the 36-cu-ft capacity.

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Discussion of the Problem

In the research program described here, the effort would be directed toward developing and evaluating an experimental full-scale generator based on the design which was evolved under Phase I (Task Order No. C).

The principal problem involved in applying the results of the small-scale studies of the sodium borohydride hydrolysis to a prototype full-scale generator is to establish the time required for complete generation. The cobalt chloride - sodium borohydride hydrolysis reaction that would be used for the proposed generator is a heterogeneously catalyzed reaction. As a result, the chemical kinetics, and therefore the total generation time, are strongly influenced by the distribution of the catalyst throughout the system. It was proposed in the Summary Report on Task Order No. C, dated January 28, 1957, that the cobalt chloride solution be introduced through 10 openings in the proposed full-scale reactor. Because of the sensitivity of this system to the dispersion of the catalyst, it is possible that 10 openings would not be sufficient. If this factor were to be scaled up directly from the Phase I research, about 100 openings would be necessary; it is not expected that such a large number of openings is required. The number of openings would be established under the proposed research program.

The rate of solution of the sodium borohydride and the homogeneity of the resulting solution are not expected to present major problems, but should be given some consideration. It is possible that, in running a unit like the proposed generator, the operator might drop a large batch of sodium borohydride in the pool within the unit, and, because of insufficient stirring, a large clump might result that would not dissolve at a rapid enough rate.

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If the reaction was started before the borohydride was completely dissolved, the hydrogen generation rate would initially be slow. It is recognized that the process of solution would continue and that the stirring resulting from the hydrogen evolution would aid in achieving complete solution of the borohydride. However, the over-all effect is uncertain.

Proposed General Method of Procedure

The proposed research program as outlined in this section has been set up to include the investigation of the effects of catalyst dispersion and of the solution rate of the borohydride on the operation of an experimental full-scale generator. Because of the cost of the sodium borohydride, each full-scale experiment would involve approximately \$3,000 for materials alone. Furthermore, if a full-scale experiment aborted, the materials involved could not be reclaimed. In an effort to reduce the cost of evaluation, and thus of the proposed program, it is contemplated that two sizes of experimental generators would be used for these studies: a 1/5-scale and a full-scale experimental generator. The smaller unit would have one-fifth the pool area and the same depth (1-1/2 ft) as the experimental full-scale generator. The diameter of the smaller unit would be about 3-1/2 ft and the generation capacity, about 740 cu ft of hydrogen. Since the reaction previously run in the experimental 36-cu-ft generator was almost adiabatic, it is not expected that the generation rate in these larger experimental generators would increase over that obtained in the 36-cu-ft unit.

Two series of experiments are proposed for the smaller experimental generator. One series would use the full pool depth of 18 in. The

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other would use a pool depth of 9 in. and would involve the generation of about 340 cu ft of hydrogen. The purpose of the half-depth studies would be to study the reaction in a pool in which the diameter-to-depth ratio would be nearly that of the proposed full-scale generator.

Studies With the Experimental 1/5-Scale Generator

The following variables would be investigated using the smaller experimental generator:

- (1) Homogeneity of the borohydride solution - the effect of the diameter-to-depth ratio on the solution homogeneity, and, in turn, its effect on the rate of generation.
- (2) pH - the effect of the amount of sodium hydroxide on the rate of solution and on the rate of generation.
- (3) Distribution of the catalyst - the effect of the spacing and the number of catalyst-entry openings (ports) on the rate of generation.
- (4) Initial temperature - the relationship between cobalt chloride and the initial temperature, and its effect on the rate of generation. (See Table 16 of the Summary Report on Task Order No. C, dated January 28, 1957.)

Since the above variables are interdependent, it is difficult to indicate accurately, at this time, the number of experiments that would have to be conducted. However, it is currently estimated that 10 to 15 experiments in the smaller experimental generator would be sufficient to provide the needed information. Some of these experiments would be run at full pool depth, and the remainder at half pool depth. Efforts would be made to

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design and conduct these experiments so as to obtain the needed information from the smallest number of experiments.

The effect of solution rate and/or clumping of the sodium borohydride would also be studied. It is expected that the prosecution of this study would not influence the total number of experiments required, since this study would represent a preliminary step in the performance of the above-indicated experiments and would be done in conjunction with them.

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Study With the Experimental Full-Scale Generator

It is believed that, at a minimum, two experiments should be run in order to investigate the operation of the experimental full-size generator. However, it may be desirable to make some minor changes in the system on the basis of the results obtained from the first full-scale experiment. Consequently, it appears prudent to provide for up to three full-scale experiments in the proposed program.

Experimental Procedure

At the start of the proposed research period, a fixed-price purchase order would be placed with [REDACTED]

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[REDACTED] for an experimental 1/5-scale generator. The specifications for this unit would be scaled down from the experimental design described in the Summary Report on Task Order No. C, dated January 28, 1957.

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The experimental full-scale generator would not be ordered at that time, since its specifications would not be established until some time after the 1/5-scale studies were under way or until these studies were concluded. It should be possible to firm up the design and place a fixed-price purchase order for the experimental full-scale generator early enough in the proposed program so as to prevent undue delay.

Consideration would be given to accessory techniques and/or minor equipment needed to facilitate the operation of the proposed experimental generators. The operational aspects that would be scrutinized would include, for example, the inflation of the experimental units preparatory to generating hydrogen and the pre-mixing of the catalyst solution.

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Because of the physical size of the experimental units and the safety hazards associated with the large amounts of hydrogen involved, it is proposed that the above-indicated experiments be conducted outdoors. However, a special building is available that would permit the 1/5-scale studies to be conducted indoors, if necessary. It is contemplated that all of the chemicals would be used as supplied by the commercial sources and that the water involved would be obtained from the rivers in the area.

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In a typical experiment, the required amount of water would be admitted into the experimental generator through the unsipped opening, the water would be made alkaline with sodium hydroxide, and then the sodium borohydride would be dissolved. The experimental unit would be sipped closed, the pre-mixed cobalt chloride solution would be added, and the volume of evolved hydrogen would be measured. In the experimental 1/5-scale generator studies, it is contemplated that the evolved hydrogen would be metered through a gas meter. The Sponsor would probably be interested in having the full-scale experiments conducted so that the hydrogen generated would be trapped in a representative balloon. Otherwise, the hydrogen from the full-scale experiments would also be metered through a gas meter. It is expected that the Sponsor would be present during at least one of the full-scale experiments.

After the conclusion of the full-scale experiments, the prototype full-scale generator would be shipped to the Sponsor for field testing. As a result of experience with the experimental full-scale unit,

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the Sponsor might have comments with regard to the unit. These would be discussed; to the extent possible within the time and funds provided in the proposed contract and within the limits imposed by the materials and method of fabrication used in the experimental full-scale unit, mutually agreed upon modifications would be made in the design. It is recommended that any other modifications be made as part of a Phase III activity, to be provided for under another contractual arrangement. Such a Phase III effort might also include the preparation of specifications, drawings, and an operator's manual, if desired.

On the basis of recent discussions, it is expected that one or two balloons, for use in the full-scale experiments, and 500 pounds of sodium borohydride would be furnished by the Sponsor. The indicated amount of borohydride is based on the requirements of the currently planned schedule of experiments, including approximately ten 1/5-scale, half-pool-depth experiments (about 10 pounds per experiment); about five 1/5-scale, full-pool-depth experiments (about 20 pounds per experiment); and up to three full-scale experiments (about 100 pounds per experiment).

Since it is important that the necessary purchase orders be placed as quickly as possible, it is recommended that the proposed contract contain express approval for the procurement of the experimental 1/5- and full-scale generators. As an alternative, the Contracting Officer could grant this approval in an accompanying letter. On the basis of preliminary discussions with it is currently

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estimated that the two experimental units would cost approximately \$6,500.

It is anticipated that a two- to three-month delivery time would be involved in the procurement of the experimental units from the commercial fabricator. It is possible that additional delays occasioned by weather interference in the scheduling of some of the proposed experiments might also be encountered. However, every effort would be made to conclude the proposed research program as quickly as possible.

Reports and Liaison

The Sponsor would be kept informed of the progress of the research program by informal monthly reports. These reports would be supplemented by meetings with the Sponsor. At the conclusion of the proposed research period, a summary report would be prepared that would include a description of the experiments conducted and the results obtained.

Duration and Estimated Costs

It is proposed that the contract provide for an eight-month period of research, with an estimated appropriation of \$18,285, including the fixed fee. The general breakdown of the estimated costs is attached.

The Contract

The proposed contract would be a period-basis research agreement, consistent with our current contractual arrangements and providing only for a fixed period of research leading toward the objective outlined in this proposal.

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